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IS 13320 (1992): Determination of iron by atomic absorption spectrophotometry - Test method [CHD 1: Inorganic Chemicals]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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भारतीय मानक
परमाण्वीय अवशोषण स्पैक्ट्रम प्रकाशमिति द्वारा क्रोमियम
ज्ञात करने की पद्धति

Indian Standard

DETERMINATION OF IRON BY ATOMIC
ABSORPTION SPECTROPHOTOMETRY —
TEST METHOD

UDC 543.422 : 546.72

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FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Chemical Standards Sectional Committee had been approved by the Chemical Division Council.

Atomic absorption spectrophotometry procedure is based on the principle that metallic atoms present in ground state will absorb incident radiation from the source (normally hollow cathode lamp) of the same wavelength they emit when excited. When radiations from the given excited element is passed through the flame containing ground state atoms of that element, the intensity of the transmitted radiation will decrease in proportion to the amount of the ground state atoms present in the flame. A hollow cathode lamp with the cathode made from the same element to be determined or with a suitable electron discharge lamp provides the radiation. The metal atoms to be measured are placed in the beam of radiation by aspirating the sample solution into the flame. A monochromator isolates the characteristic radiations from the hollow cathode lamp and photosensitive device measures the attenuated transmitted radiation. The absorbance is taken as the measure of concentration of the analysed element. Various metallic elements may be precisely analysed by this technique, and is generally adopted for estimation of iron in samples like water, brines, mineral deposits, ores, pickling solutions, etc.

In the preparation of this standard considerable assistance has been derived from ASTM-D 1068 'Test methods for iron in water' published by the American Society for Testing and Material, USA.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

DETERMINATION OF IRON BY ATOMIC ABSORPTION SPECTROPHOTOMETRY — TEST METHOD

1 SCOPE

This standard prescribes method for determination of iron by atomic absorption spectrophotometry.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard :

IS No.	Title
264 : 1976	Specification for nitric acid (<i>second revision</i>)
265 : 1987	Specification for hydrochloric acid (<i>third revision</i>)
1070 : 1977	Specification for water for general laboratory use (<i>second revision</i>)

3 OUTLINE OF THE METHOD

The sample is brought into solution by suitable treatment with acid, diluted with distilled water, filtered if necessary. Further dilution may be made depending on the concentration required. The samples are then aspirated in an air-acetylene flame. The most sensitive wavelength for iron is 248.3 nm. However, other wavelengths may also be used for higher concentrations. These wavelengths are generally indicated in apparatus.

4 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

5 INTERFERENCE

5.1 Sodium, potassium, barium, chloride and sulphate in concentrations less than 5 000 mg/l each, calcium, magnesium, chromium, manganese, cobalt, nickel, copper, zinc, palladium, silver, cadmium, tin, lead, lithium, mercury, selenium, aluminium, antimony, arsenic, vanadium, boron, molybdenum in concentrations less than 100 mg/l each do not interfere.

5.2 Background correction by techniques, such as a continuum source, non-absorbing lines, or chelation-extraction may be necessary to determine low levels of iron in some water.

6 APPARATUS

6.1 Atomic Absorption Spectrophotometer, for use at 248.3 nm.

NOTE — The manufacturer's instructions shall be followed for all instrumental parameters.

6.2 Iron Hollow-Cathode Lamp

Multi-component hollow cathode lamps may be used.

6.3 Pressure Reducing Valves

Suitable for flow control of fuel.

6.4 Wavelength and Working Range

In addition to the wavelength 248.3 nm, the following wavelengths and their working range may be used:

Sl No.	Wavelength (nm)	Slit (nm)	Working Range (mg/l)	Sensitivity (mg/l)
(1)	(2)	(3)	(4)	(5)
i)	372.0	0.2	25 to 100	0.55
ii)	386.0	0.2	50 to 200	0.90
iii)	392.0	0.2	800 to 3 200	17

7 REAGENTS

7.1 Concentrated Hydrochloric Acid — *See* IS 265 : 1987.

7.2 Nitric Acid — *See* IS 264 : 1976.

7.3 Dilute Nitric Acid — 0.2 percent (v/v).

Add 1 volume of nitric acid (sp. gr. 1.42) to 499 volumes of water.

7.4 Stock Iron Solution

Dissolve 1.000 g of pure iron in 100 ml of hydrochloric acid (1:1) by heating. Cool and dilute to 1 000 ml with water. 1 ml = 1.0 mg of iron.

7.5 Standard Iron Solution

Dilute 100 ml of the iron stock solution to 1 litre with water. 1 ml = 0.1 mg of iron.

7.6 Oxidant : Air

Which has been passed through a suitable filter to remove oil, moisture and other foreign substances.

7.7 Fuel : Acetylene

Commercially available acetylene is the usual fuel.

8 PROCEDURE

8.1 Sample Preparation

8.1.1 Water Samples

Measure a suitable volume of sample in a beaker or conical flask. Add 50 ml of water. Add 5 ml of hydrochloric acid (sp gr 1.19). Heat the sample on a steam bath or hot plate in a well ventilated hood. Reduce the volume to 15 ml. Cool the solution and filter through a Whatman No. 41 or 42 filter paper. Collect filtrate in 100 ml volumetric flask. Wash the filter with water and collect washings in the same volumetric flask. Make up to 100 ml.

8.1.2 Solid Samples (Ores, Mineral Deposits, etc)

Powder the sample with the help of a clean and dry pestle and mortar. Transfer a known amount of the sample to a clean and dry porcelain or quartz crucible. Fuse this material at about 700 to 800°C with a minimum of potassium or sodium bisulphate (KHSO_4 or NaHSO_4). Cool the crucible to room temperature and add 50 ml of water and 2 ml of hydrochloric acid (sp gr 1.19). At this stage if the solution is not clear, then concentrate the solution to about 10 ml and add

3 ml of nitric acid (sp. gr. 1.42), 3 ml of perchloric acid (70 percent) and 3 ml of sulphuric acid (sp gr 1.84). Evaporate to dense white fumes, cool the beaker and wash the inside of the beaker with water. Add a few drops of formic acid and fume again to dense cloud. Cool and add water carefully. Make up to a suitable volume.

8.2 Analysis

Aspirate the standard iron solutions and then sample solutions. Aspirate dilute nitric acid (1 : 499) between each standard and sample. Choose the standards in such a way that the absorbance of sample solution is within the range of calibration.

Plot a graph for standards versus absorbances and find out microgram per millilitre of iron in sample solution.

9 CALCULATION

$$\text{Iron, percent by mass} = \frac{C \times V}{10^6} \times \frac{100}{M}$$

where

- C = concentration of iron in $\mu\text{g/ml}$ in final solution,
- V = volume in ml of the final solution, and
- M = mass in g of the sample in final solution.

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